## => d his; log y

(FILE 'HOME' ENTERED AT 16:55:12 ON 21 JUL 2006)

FILE 'CAPLUS' ENTERED AT 16:55:26 ON 21 JUL 2006

L1 96 S CAPROLACTA? AND (AMMONIA OR AMONI?) AND WATER?

L2 15 S L1 AND DISTIL?

COST IN U.S. DOLLARS

SINCE FILE TOTAL
ENTRY SESSION
FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

CA SUBSCRIBER PRICE

SINCE FILE TOTAL
ENTRY SESSION
-11.25

STN INTERNATIONAL LOGOFF AT 16:57:27 ON 21 JUL 2006

- L2 ANSWER 1 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 2006:669703 CAPLUS Full-text
- TI Hydroxylamine sulfate production process
- IN Mamedov, A. A.; Barabash, I. I.; Konoplina, O. V.
- PA Ukrainskii Gos. Nauchno-Issledovatel'skii i Proektnyi Inst. Azotnoi Promyshlennosti i Produktov Org. Sinteza, Ukraine
- SO Russ., 5 pp. CODEN: RUXXE7
- DT Patent
- LA Russian

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PΙ	RU 2279401	C2	20060710	RU 2004-122497	20040722
PRAI	UA 2003-87439	Α	20030806		

AB FIELD: industrial organic synthesis.SUBSTANCE: hydroxylamine sulfate (starting material in production of caprolactam) production process comprises: preparing reaction mixture of ammonia, oxygen, and water steam; catalytically oxidizing ammonia at pressure above 0.3 MPa; stabilizing composition of nitrose gas via hydrogenation of silver-manganese catalyst; concentrating nitrose gas by means of water steam condensation; combining concentrated nitrose gas with hydrogen and mixture of sulfuric acid, distillate produced in nitric acid condensate concentration, a part of secondary steam condensate produced during concentration of hydroxylamine sulfate, and catalysate remaining after catalytic hydrogenation of nitric acid contained in concentrated nitric acid condensate; catalytically hydrogenating nitric acid; and synthesis of hydroxylamine sulfate. Invention is characterized by that, (i) for concentration of nitric acid condensate to weight percentage of nitric acid 8%, heat of water steam condensation proceeding during concentration of nitrose gas is used; and (ii) hydroxylamine sulfate solution is concentrated to weight percentage 38% using heat of condensation of distillate vapors produced during concentration of nitric acid condensate in distillation column.EFFECT: reduced power consumption at the same specific intake of raw materials.1 dwg, 1 tbl.

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L2
    ANSWER 2 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
ΑN
    2005:1314289 CAPLUS Full-text
DN
    144:52059
ΤI
    Method for separating ammonia and water from mixtures,
    arising during the production of polyamides.
IN
    Assmann, Jens; Demeter, Juergen; Deininger, Juergen; Soetje, Oliver; Kory,
    Gad; Loening, Jan-Martin
PΑ
    Basf Aktiengesellschaft, Germany
SO
    PCT Int. Appl., 22 pp.
    CODEN: PIXXD2
DT
    Patent
LA
    German
FAN.CNT 1
    PATENT NO.
                       KIND
                             DATE APPLICATION NO.
                                                                DATE
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                                        -----
                               20051215 WO 2005-EP5833
PΙ
    WO 2005118692
                        A1
                                                               20050531
        W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,
            CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,
            GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KP, KR, KZ,
            LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA,
            NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK,
            SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU,
            ZA, ZM, ZW
        RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
            AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,
            EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT,
            RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML,
            MR, NE, SN, TD, TG
    DE 102004027022
                        Α1
                               20060105
                                          DE 2004-102004027022
                                                                 20040602
PRAI DE 2004-102004027022 A
                               20040602
     A method for the distillative separation of ammonia and water from mixts.
     arising during the production of polyamides comprises 2 steps: (a) the mixture
     is distilled at 180 - 2600° and 11 - 35 bars, resulting in the production of
     water, ammonia and cyclopentanone as overhead products (K1), and water, lactam
     and/or diamines and, optionally, aminonitrile and/or dinitrile, as base
     products (S1), and (b) K1 is distilled at 11 - 35 bars, resulting in the
     production of ammonia and cyclopentanone as an overhead product, and water as
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RE.CNT 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

a base product (S2).

- L2 ANSWER 3 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 2005:1197875 CAPLUS Full-text
- DN 143:406259
- TI Method for obtaining high-purity caprolactam
- IN Danilczyk, Natalia; Gucwa, Antoni Janusz; Gwizdak, Marek; Izydorczyk, Kazimierz; Kania, Jan; Lonak, Boguslaw; Maciszewski, Leszek; Maczuga, Jan; Makal, Konstanty; Malinowska, Magdalena; Rygiel, Stanislaw; Szparski, Jozef; Wais, Jan
- PA Zaklady Azotowe w Tarnowie-Moscicach SA, Pol.; Biuro Projektow Zakladow Azotowych Biprozat-Tarnow Sp.z oo.
- SO Pol., 6 pp. CODEN: POXXA7
- DT Patent
- LA Polish

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE	
PΙ	PL 188522	В1	20050228	PL 1997-321644	19970813	
PRAI	PL 1997-321644		19970813			

AB High-purity caprolactam is prepared by the Beckmann rearrangement of cyclohexanone oxime in the presence of oleum, the mixture is neutralized with aqueous ammonia, extracted with trichloroethylene and water , distilled, and crystallized

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L2 ANSWER 4 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
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AN 2005:140974 CAPLUS Full-text

DN 142:200998

- TI Method for producing hydroxylamine sulfate
- IN Mamedov, Abil Abasovich; Barabash, Ivan Ivanovich; Konoplina, Olga Viktorovna
- PA Ukrainian State Scientific and Research Institute of Nitric Industry and Organic Synthesis Products UKRGIAP, Ukraine
- SO PCT Int. Appl., 12 pp. CODEN: PIXXD2

DT Patent

LA Russian

FAN.CNT 1

	01.1 1																
	PATENT	KIN	KIND DATE			i	APPL	ICAT		DATE							
						_									_		
ΡI	WO 2005014473			A1 20050217			WO 2004-UA31						20040518				
	W:	ΑE,	AG,	AL,	AM,	AT,	ΑU,	AZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,	CA,	CH,
		CN,	CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,	ES,	FI,	GB,	GD,
		GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	ΚE,	KG,	ΚP,	KR,	KZ,	LC,
		LK,	LR,	LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	ΜZ,	NA,	NI,
		NO,	NZ,	OM,	PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	SY,
		ТJ,	TM,	TN,	TR,	TT,	ΤZ,	UA,	UG,	US,	UZ,	VC,	VN,	YU,	ZA,	ZM,	ZW
	RW:	BW,	GH,	GM,	ΚE,	LS,	MW,	MZ,	NA,	SD,	SL,	SZ,	ΤZ,	UG,	ZM,	ZW,	AM,
		ΑZ,	BY,	KG,	ΚZ,	MD,	RU,	ТJ,	TM,	ΑT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,
		EE,	ES,	FI,	FR,	GB,	GR,	HU,	ΙE,	ΙT,	LU,	MC,	NL,	PL,	PT,	RO,	SE,
		SI,	SK,	TR,	BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	ΝE,
		SN.	TD.	TG													

PRAI UA 2003-87438 A 20030806

AB Hydroxylamine sulfate (HAS) is produced by preparing a reaction mixture containing ammonia, oxygen, and steam, catalytically oxidizing ammonia at > 0.3 MPa, concentrating produced NO by condensating the water vapor, mixing NO with H2, a mixture of sulfuric acid, water, and the condensate of the concentrated NO to produce HAS. The heat of the NO is used for concentrating the nitric acid containing condensate and the HAS solution. The nitric acid condensate is concentrated until a nitric acid mass fraction of > 40% is obtained which can be used for producing mineral fertilizers. The HAS solution is concentrated until a mass fraction of > 40% is obtained using the distillate condensing heat produced during nitric acid condensate concentration. The produced HAS can be used for caprolactam production.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

```
ΑN
     2004:1127152 CAPLUS Full-text
DN
     142:74968
TΙ
     Hydrolytic and distillation method for making \epsilon-
     caprolactam from impure 6-aminocapronitrile in which
     tetrahydroazepine is not removed until after the \epsilon-
     caprolactam is produced
IN
     Kirby, Gregory S.; Ostermaier, John J.
     Invista North America S.A.R.L., USA
PA
     U.S. Pat. Appl. Publ., 6 pp.
SO
     CODEN: USXXCO
DT
     Patent
LA
     English
FAN.CNT 1
     PATENT NO.
                       KIND
                                DATE
                                           APPLICATION NO.
                                                                   DATE
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                                -----
                                            _____
PΙ
     US 2004260087
                         A1
                                20041223
                                            US 2003-464104
                                                                   20030617
     US 6858728
                         B2
                                20050222
     WO 2005000808
                         A1
                                20050106
                                            WO 2004-US19442
                                                                   20040617
            AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,
             CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,
             GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,
             LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI,
             NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY,
             TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
         RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
             AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,
             EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
             SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,
             SN, TD, TG
     EP 1636179
                         A1
                                20060322
                                            EP 2004-785751
                                                                   20040617
         R: BE, DE, FR, NL
PRAI US 2003-464104 A
                                20030617
     WO 2004-US19442
                         W
                                20040617
     A method for making caprolactam from an impure 6-aminocapronitrile (ACN),
AΒ
     obtained by the partial hydrogenation of adiponitrile, which comprises 6-
     to produce a vapor-phase reaction product that comprises &- caprolactam,
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ANSWER 5 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN

L2

aminocapronitrile and both ACN and ≥500 ppm tetrahydroazepine and its derivs. (THA), comprises: (1) contacting the impure ACN comprising both ACN and THA with water at elevated temperature in the presence of a dehydration catalyst (e.g., alumina), both the impure ACN and the water being in the vapor phase, ammonia, water, ACN, and THA; (2) separating the ammonia and a major portion of the water from the vapor-phase reaction product to produce a solution comprising  $\epsilon$ -caprolactam and a minor portion of the water , and then separating the water from the solution to produce a melt comprising &caprolactam, ACN and THA; (3) introducing the melt into a low-boiler-removal distillation column and removing a major portion of both the THA and ACN as a distillate, and removing &-caprolactam, high boilers and at most a minor portion of both the THA and ACN as a bottoms; and (4) introducing the bottoms into a high-boiler-removal distillation column and removing &-caprolactam and at most a minor portion of the high boilers as a distillate product and removing a major portion of the high boilers as a bottoms. Process flow diagrams are presented.

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

App's

- L2 ANSWER 6 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 2004:428902 CAPLUS Full-text
- DN 140:407268
- TI Purification of caprolactam
- IN Fischer, Rolf-Hartmuth; Luyken, Hermann; Ansmann, Andreas; Bassler, Peter; Benisch, Christoph; Maixner, Stefan; Melder, Johann-Peter
- PA BASF Aktiengesellschaft, Germany
- SO PCT Int. Appl., 27 pp. CODEN: PIXXD2
- DT Patent
- LA German

FAN. CNT 1

FAN.	CNT	1																	
	PATENT NO.					KIND DATE			APPLICATION NO.						DATE				
ΡI	WO	2004	0439	14		A1		2004	0527	1						20	0031	111	
		W:	ΑE,	AG,	AL,	AM,	AT,	ΑU,	AZ,	BA,	BB,	BG,	BR,	BY,	ΒZ,	CA,	CH,	CN,	
								DK,											
			GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	ΚE,	KG,	KP,	KR,	ΚZ,	LC,	LK,	
			LR,	LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NI,	NO,	NZ,	
			OM,	PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	SY,	ТJ,	TM,	
			TN,	TR,	TT,	TZ,	UA,	UG,	US,	UZ,	VC,	VN,	YU,	ZA,	ZM,	zw			
		RW:	BW,	GH,	GM,	ΚE,	LS,	MW,	ΜZ,	SD,	SL,	SZ,	TZ,	UG,	ZM,	ZW,	AM,	AZ,	
			BY,	KG,	KZ,	MD,	RU,	ТJ,	TM,	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	
			ES,	FI,	FR,	GB,	GR,	HU,	ΙE,	IT,	LU,	MC,	NL,	PT,	RO,	SE,	SI,	SK,	
			TR,	BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	NE,	SN,	TD,	TG
	DE	1025	3095			A1 20040617				DE 2002-10253095						20021113			
	CA	2505	356			AA		2004	0527	CA 2003-2505356						20031111			
	ΑU	2003	2820	93		A1		2004	0603	AU 2003-282093						21	0031	111	
	ΕP	1562	896			A1		2005	0817		EP 2	003-	7737	10		21	0031	111	
		R:	ΑT,	BE,	CH,	DE,	DK,	ES,	FR,	GB,	GR,	ΙT,	LI,	LU,	NL,	SE,	MC,	PT,	
			ΙE,	SI,	LT,	LV,	FI,	RO,	MK,	CY,	AL,	TR,	BG,	CZ,	EE,	HU,	SK		
	BR	2003	0161	78		Α		2005	0927		BR 2	003-	1617	8		2	0031	111	
	JP	2006	5081	15		T2		2006	0309		JP 2	004-	5509	78		2	0031	111	
	US	2006	0411	22		A1		2006	0223	1	US 2	005-	5348	02		2	0050	513	
PRAI	DE	2002	-102	5309	5	Α		2002	1113										
	WO	2003	-EP1	2556		W		2003	1111										

The invention relates to a method for separating high-boiling material from crude caprolactam, which contains high boilers, caprolactam and, optionally, low boilers, and which has been obtained by: (a) reacting 6-aminocapronitrile with water to form a reaction mixture and (b) separating ammonia and unreacted water out from the reaction mixture while obtaining the crude caprolactam. The invention is characterized in that: (c) the crude caprolactam is fed to a distillation device while obtaining, as a product, a first partial stream via the top, and obtaining a second partial stream via the bottom. During distillation, the pressure is set so that a bottom temperature of at least 170°C is maintained, and the second partial stream is set so that the caprolactam content of the second partial stream is no less than 10 % by weight with regard to the entire second partial stream.

RE.CNT 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

```
ΑN
     2004:282869 CAPLUS Full-text
DN
    140:304207
ΤI
    Method for making caprolactam from impure 6-aminocapronitrile
    where ammonia and water are removed from crude
     caprolactam in a simple separation step and then tetrahydroazepine
    and its derivatives are removed from the resulting caprolactam
    melt
    Kirby, Gregory S.; Ostermaier, John J.
ΙN
PA
    E. I. Du Pont De Nemours and Company, USA
    U.S., 6 pp.
SO
    CODEN: USXXAM
DT
    Patent
LA
    English
FAN.CNT 1
     PATENT NO.
                       KIND
                               DATE
                                         APPLICATION NO.
                                                                  DATE
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                               -----
                                           -----
                                                                 _____
    US 6716977
                         В1
                                        US 2003-464175
PΙ
                               20040406
                                                                  20030617
    WO 2004113288
                        A1
                               20041229 WO 2004-US19432
                                                                  20040617
        W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,
            CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,
            GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,
            LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI,
            NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY,
            TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
        RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
            AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,
            EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
             SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,
            SN, TD, TG
    EP 1636178
                         A1
                               20060322 EP 2004-755552
                                                                  20040617
        R: BE, DE, FR, NL
PRAI US 2003-464175
                     Α
                               20030617
    WO 2004-US19432
                        W
                               20040617
AΒ
     A method for making caprolactam from an impure 6-aminocapronitrile (ACN) that
     comprises both ACN and a min. of 500 ppm tetrahydroazepine and its derivs.
     (THA), comprises: (1) contacting the impure ACN comprising both ACN and THA
     with water at elevated temps. in the presence of a dehydration catalyst, both
     the impure ACN and the water being in the vapor phase, to produce a vapor-
     phase reaction product that comprises caprolactam, ammonia, water, ACN, and
     THA; (2) separating the ammonia and a major portion of the water from the
     vapor-phase reaction product to produce a melt comprising caprolactam, ACN and
     THA; (3) introducing the melt into a low-boiler removal distillation column
     and removing a major portion of both the THA and ACN as a distillate , and
     removing caprolactam, high boilers and at most a minor portion of both the THA
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and ACN as a bottoms product; and (4) introducing the bottoms product into a high-boiler removal distillation column and removing caprolactam and at most a minor portion of the high boilers as a distillate product and removing a major portion of the high boilers as a bottoms product. Process flow diagrams are

ANSWER 7 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN

L2

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

presented.

- L2 ANSWER 8 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 2000:350321 CAPLUS Full-text
- DN 132:322256
- TI Process for separation of caprolactam from caprolactam sulfate
- IN Lucasevici, Traian; Jumanca, Valeriu; Corchez, Aurora
- PA S.C. Fibrex S.A., Savinesti, Rom.
- SO Rom., 4 pp. CODEN: RUXXA3
- DT Patent
- LA Romanian

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PΙ	RO 108682	B1	19940729	RO 1991-147760	19910612
PRAI	RO 1991-147760		19910612		

AB In the manufacture of caprolactam by Beckmann rearrangement of cyclohexanone oxime with H2SO4, the lactam (as the sulfate salt) is extracted with benzene or trichloroethylene, the extract is treated with NH3 and sufficient H2O to decompose the salt, the (NH4)2SO4 formed is removed by filtration or centrifugation, and the caprolactam is recovered by distillation. The weight ratio of solvent:caprolactam sulfate:water is 1-3:1:0.1-0.3.

- L2 ANSWER 9 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 2000:84721 CAPLUS Full-text
- DN 132:137831
- TI Method for ammonia distillation in caprolactam manufacture
- IN Bocquenet, Gerald; Houssier, Patrick
- PA Rhodia Fiber and Resin Intermediates, Fr.
- SO PCT Int. Appl., 13 pp. CODEN: PIXXD2
- DT Patent
- LA French
- FAN.CNT 1

PAN.																				
									TE APPLICATION NO.								ATE			
ΡI									WO 1999-FR1731											
		W:		BY,				ID,												
		RW:		BE, SE		CY,	DE,	DK,	ES,	FI,	FR	ι, ο	GΒ,	GR,	IE,	IT,	LU,	MC,	NL,	
	FR	2781	476			A1		2000			FR	199	98-	9530			1	9980	722	
		<ul><li>2781</li><li>2337</li></ul>						2000			CA	199	99-:	2337	321		1	9990	715	
	CA	2337	321			С		2003	0211											
		1102 1102						2001			ΕP	199	99-	9294	90		1	9990	715	
		R:	AT,		CH,						GR	R, I	ΙΤ,	LI,	LU,	NL,	SE,	MC,	PT,	
		9912	346			Α		2002	0115		BR	199	99-:	1234	6		1	9990	715	
	JP	2002	5213	49		Т2		2002	0716		JP	200	00-	5611	31		1	9990	715	
	RU	2186	026			C1		2002	0727		RU	200	01-1	1048	97		1	9990	715	
	ΑT	2354	23			E		2003	0415						90			9990	715	
		2190						2003	0716		ES	199	99-	9294	90		1	9990	715	
		2837				В6		2003	1202											
		4692						2001							2391					
		6482						2002			US	200	01-	7441	56		2	0010	606	
PRAI		1998						1998												
	MO	1999	-FR1	731		W		1999	0715											

AB The invention concerns an improved method for NH3 distillation from a mixture, especially a mixture derived from the reaction between an aminonitrile and water (i.e., cyclizing hydrolysis reaction). The invention concerns a method for distillation of NH3 contained in an aqueous caprolactam solution. The distillation is carried out in a column having a bottom temperature of  $\leq 160^{\circ}$  and pressure  $\leq 5$  bar; the NH3 is withdrawn at the column top at  $\geq 10$  bar and then condensed at  $25-60^{\circ}$ .

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

```
L2 ANSWER 10 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
```

AN 1996:191605 CAPLUS Full-text

DN 124:290535

TI Preparation of caprolactam

IN Achhammer, Guenther; Fuchs, Eberhard

PA BASF A.-G., Germany

SO U.S., 3 pp.

CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

L MIV.		_																	
	PATENT NO.					KIND DATE		APPLICATION NO.						Di	ATE				
ΡI	US 5495016						1996	0227	- T	IS 1	991-	3584	 1 1		1 4	99/1	210		
		4441									-								
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		3792						2000	0111	7	CW 1:	995-8	3410	6025		1:	9950	513	
	WO	9616	936			A1		1996	0606	V	VO 1	995-I	EP44	64		1:	19951114		
		W:	ΑU,	BG,	BR,	BY,	CA,	CN,	CZ,	FI,	HU,	JP,	KR,	ΚZ,	MX,	NO,	ΝZ,	PL,	
			RU,	SG,	SK,	UA													
		RW:	AT,	BE,	CH,	DE,	DK,	ES,	FR,	GB,	GR,	ΙE,	ΙΤ,	LU,	MC,	NL,	PT,	SE	
	ΑU	9539	287			A1		1996	0619	I	AU 1	995-3	3928	7		1:	9951	114	
	ΕP	7936	50			A1		1997	0910	E	EP 1	995-9	9370	70		1:	9951	114	
	EΡ	7936	50			В1		2002	0703										
		R:	AT,	BE,	CH,	DE,	DK,	ES,	FR,	GB,	GR,	ΙE,	IT,	LI,	LU,	MC,	NL,	PT,	SE
	CN	1166	830			Α		1997	1203	(	CN 1	995-3	1964	00		1	9951	114	
	CN	1070	182			В		2001	0829										
	JP	1050	9963			Т2		1998	0929	j	JP 1:	995-5	5181	23		1:	9951	114	
	RU	2153	492			C2		2000									9951		
	ΑT	2200	61			E		2002	0715	I	AT 1	995-9	9370	70		1	9951	114	
	CZ	2910	34			В6		2002	1211	(	CZ 1	997-:	1469			1	9951	114	
	ES	2179	888			Т3		2003	0201	E	ES 1	995-9	9370	70		1	9951	114	
PRAI	DE	1994	-444	1962		Α		1994	1125										
	WO	1995	-EP4	464		W		1995	1114										

AB Caprolactam is obtained with high selectivity and in high yield starting from 6-aminocapronitrile in the liquid phase, without a catalyst, in short reaction times. A solution of 10% 6-aminocapronitrile in water was heated to 300°, the product mixture (mixture A) contained 90% water and 10% a mixture containing 76% caprolactam and 24% high boilers, this mixture A was then distilled at 100-300 mbar in a column having 5 theor. plates, giving ammonia-containing water as the top product and caprolactam and the high boilers at the bottom of the column (mixture B), then mixture B was separated at 3-10 mbar into a caprolactam fraction (top product) and a high-boiling fraction (bottom product), giving 74% caprolactam.

- L2 ANSWER 11 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 1994:84836 CAPLUS <u>Full-text</u>
- DN 120:84836
- TI Polymer-mineral mixtures
- IN Samigov, Nigmatdzhan A.; Solomatov, Vasilij I.; Dzhalilov, Abdulakhat T.;
  Usmanov, Said Akrom N.; Fatkhullaev, Erkinzhon; Khabilov, Nadir B.;
  Kuchkarov, Khusan Ya
- PA Tashkent Polytechnic Institute, USSR
- SO U.S.S.R.

From: Izobreteniya 1992, (15), 90.

CODEN: URXXAF

DT Patent

LA Russian

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	SU 1728164	A1	19920423	SU 1990-4805730	19900117
PRAI	SU 1990-4805730		19900117		

The mixts. comprise H2CO-urea resin 31-32, plaster of Paris from phosphogypsum 63-65, reaction products of polyethylenepolyamine with NaSCN or KCNS in equimol. ratio 0.93-0.96, and superplasticizer 0.25-0.45 weight%, and balance water. The plasticizer is the reaction product of distillation residues from the manufacture of benzoic acid by PhMe oxidation (A), distillation residues from the purification of NH3 manufactured from monoethanolamine (B), and wastewater containing Na carboxylates from the manufacture of caprolactam from PhMe (C) in A/B/C weight ratio 50:11.5:61.5.

- L2 ANSWER 12 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 1986:225351 CAPLUS Full-text
- DN 104:225351
- TI  $\epsilon$ -Aminocaproic acid by hydrolysis of  $\epsilon$ -caprolactam
- IN Winzer, Werner; Hofmann, Guenter; Schuetze, Ralf; Pester, Rolf; Raese, Hasn Eberhard
- PA VEB Leuna-Werke "Walter Ulbricht", Ger. Dem. Rep.
- SO Ger. (East), 12 pp. CODEN: GEXXA8

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DD 217209	A1	19850109	DD 1983-253547	19830801
PRAI	DD 1983-253547		19830801		

AB An aqueous ε-caprolactam (I) solution containing ≥1 mol NH3/mol I is heated ≤20 h at 120-170°, cooled rapidly to <105°, freed of NH3 by distillation, and freed of unreacted I (especially after concentration by distillation) by extraction, and ε- aminocaproic acid (II) is recovered from the solution by evaporation and crystallization The process gives II without excessive formation of byproducts and waste materials. Thus, 1200 parts 8.0:14.0:78 I-NH3-water mixture was heated to 140°/1.4 MPa during 5 h, and the pressure was decreased to 1 atm with evaporation of water and most of the NH3 (which was condensed and recycled). The reaction mixture, containing II 4.1, I 6.0, and water 87.4%, was distilled to give a concentrate containing II 25.9, I 36.7, and water 37.4%. The concentrate was extracted with Cl2C:CHCl to remove I. The residue was evaporated and crystallized to give 40.9 parts II (98.0% yield). The I was extracted from the Cl2C:CHCl with water and recycled. The loss of I was 1.98%.

- L2 ANSWER 13 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN
- AN 1983:540564 CAPLUS Full-text
- DN 99:140564
- TI Recovery of caprolactam from products of the Beckmann rearrangement of cyclohexanone oxime neutralized with aqueous ammonia solution
- IN Gorodetskii, I. Ya.; Vasin, A. A.; Kostanyan, A. E.; Gogoladze, G. T.;
   Kervalishvili, Z. Ya.; Pagava, G. A.; Legochkina, L. A.; Tikhonovich, E.
   S.
- PA USSR
- SO U.S.S.R.

From: Otkrytiya, Izobret., Prom. Obraztsy, Tovarnye Znaki 1983, (22), 57. CODEN: URXXAF

- DT Patent
- LA Russian

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	SU 1022967	A1	19830615	SU 1980-2999411	19800804
PRAI	SU 1980-2999411		19800804		

AB Caprolactam (I) [105-60-2] of improved quality is recovered from the title products by separating the lactam oil layer from aqueous (NH4)2SO4 solution, selectively extracting I with an organic extractant from the lactam oil and from the (NH4)2SO4 solution, treating the extract (A) from the (NH4)2SO4 solution with water [which resulted from washing the extract (B) from the I oil layer] prior to adding A to B at 1:100-1:10 ratio of the above water to A, and then distilling

L2 ANSWER 14 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN

AN 1966:27133 CAPLUS Full-text

DN 64:27133

OREF 64:4951e-q

TΙ ε-Caprolactams

IN Isard, Arsene; Lakodey, Andre; Weiss, Francis

PA Societe d'Electro-Chimie, d'Electro-Metallurgie et des Acieries Electriques d'Ugine

SO 10 pp.

DT Patent

LA Unavailable

FAN.CNT 1

AΒ  $\epsilon$ -Caprolactam (I) is prepared by the reaction of  $\epsilon$ -hydroxycaproic acid derivs. with ammonia in a pressure vessel at 140-280° with a catalyst and a suitable solvent; I is distilled, and the polymeric residue heated with ammonia and water at  $250-350^{\circ}$  in a pressure vessel and extracted with a solvent to give more I. A mixture of 655 g.  $\epsilon$ -hydroxycaproamide, 2500 g. dioxane, 850 g. ammonia, and 50 g. Raney Ni are poured into a pressure vessel and heated at 225° for 3.5 hrs. The mixture is cooled, filtered and distilled to give 151 g. ε-hydroxycaproamide, and 266 g. ε-caprolactam. The residue, 159 g. of polymeric material, is heated with 1500 g. 4% ammonia for 3 hrs. at 300° cooled and extracted with 1000 g. CHCl3; evaporation of the solvent gives 54 g. I. The aqueous solution is evaporated in vacuo, and the residue heated at 225° for 3 hrs. with 80 g. anhydrous ammonia, 400 g. dioxane, and 10 g. Raney Ni. The reaction mixture is distilled to give 48 g. I. The total yield of I is 84.5%. A complete study of the reaction conditions, and the influence of the catalysts and solvents on the yield of I is also performed.

L2 ANSWER 15 OF 15 CAPLUS COPYRIGHT 2006 ACS on STN

AN 1962:403671 CAPLUS Full-text

DN 57:3671

OREF 57:655f-i,656a-c

TI Radical reactions in solution. Haloalkylation of acrylic acid derivatives

AU Minisci, Francesco; Pallini, Ugo

CS Polytecnico, Milan, Italy

SO Gazzetta Chimica Italiana (1961), 91, 1030-6 CODEN: GCITA9; ISSN: 0016-5603

DT Journal

LA Italian

AB Some new reactions, which allowed addition of an alkyl group and a halo atom to the double bond of acrylic derivs. were described. The alkyl group derived from the decomposition of a peroxide, induced by cuprous and ferrous salts. Thus, 30 g. cyclopentanone peroxide, substantially consisting of 1-hydroxy-1'hydroperoxycyclopentyl peroxide, was added with stirring and cooling to acrylonitrile (53 g.), CuCl (20 g.), and HCl (6 g.) in water (150 ml.), maintaining the temperature at -10 to  $-5^{\circ}$ . The mixture was extracted with diethyl ether, the extract treated with NaHCO3 solution, the alkaline solution treated with HCl, the oil extracted with Et2O, the extract evaporated, the residue dissolved in MeOH(100 ml. containing, 5 g. H2SO4), and refluxed 3 hrs. The solvent was removed, the residue neutralized, the oil washed with water, and distilled under reduced pressure to give at 95°/0.4 mm. methyl  $\delta$ chlorovalerate and at 112-18 $^{\circ}/0.4$  mm. methyl  $\alpha$ -chlorosuberate (I). I refluxed 5 hrs. with 100 ml. HCl concentrated gave  $\alpha$ -chlorosuberic acid (II), m. 99-100°(C6H6). II (5.7 g.) in NaHCO3 solution (6.9 g. in 150 ml. H2O) was hydrogenated in the presence of Raney Ni at 105 atmospheric at 115-20° 4 hrs., the catalyst filtered off, and the solution acidified with H2SO4 to give 4.8 g. suberic acid, m. 139-40°. Cyclohexanone (III) (9.8 g.) was dissolved in anhydrous Et20 (50 ml.) containing H2O2 (3.6 g.), the solution allowed to stand 24 hrs. at room temperature, the solvent removed, and the residue added under stirring and cooling together with CH2: CHCN (10.6 g.) to CuCl (6 g.), HCl (3 g.), and H2O (50 ml.) at  $-50^{\circ}$ . The organic layer was extracted with NaHCO3 solution, the acids obtained from the alkaline solution esterified with MeOH, and distilled to give methyl  $\alpha$ -chloroazelaate (IV), b1 125°, and methyl  $\epsilon$ -chlorocaproate (V), b16 100°. Cyclohexanone peroxide (VI), prepared as described below (6.5 g.), was added with stirring and cooling to FeSO4.7H2O(12 g.), concentrated HCl (6 ml.), and CH2:CHCN (8 g.) in water (40 ml.) at 0 to -5°. Working up as described below gave IV and V. IV hydrolyzed gave  $\alpha\text{--}$ chloroazelaic acid (VII), m. 86-7 $^{\circ}$ . VII treated as described for II gave azelaic acid (VIII), m. 107°(H2O). V (10 g.) in dioxane (450 ml.) was heated in a sealed tube at  $250-5^{\circ}$  with ammonia (25 q.) in a nitrogen atmospheric with stirring. The mixture, after cooling, was filtered, the solvent removed, and the residue distilled to give 5.7 g. caprolactam (IX), m. 68°, b0.7 99-101°. Methyl ethyl ketone peroxide (20 g.) in Et2O (15 ml.) was added with stirring and cooling to CH2: CHCN (40 g.), CuCl (25 g.), and 4 g. HCl in H2O (150 ml.) at -10 to 0°. The organic layer was decanted and fractionally distilled to give  $\alpha$ -chlorovaleronitrile, b. 160°, which by hydrolysis gave  $\alpha$ -chlorovaleric acid, b.  $221-2^{\circ}$ . Analogously,  $\alpha$ -bromovaleric acid, b0.6 84-7°, was obtained and C6-10  $\alpha$ -halo acids were prepared from Me Pr, Me Bu, Me Am, Me hexyl, Me heptyl ketones.